CONFERENCE ON DISARMAMENT

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ISLAMIC REPUBLIC OF IRAN

National Trial Inspection

INTRODUCTION

The present technical report is the outcome of the experiment carried out under the "National Trial Inspection" (NTI) at one of the chemical plants in order to contribute to the success of international efforts aimed at totally eliminating chemical weapons through a comprehensive convention of prohibition of production, stockpiling, development and use of chemical weapons. The inspection was carried out pursuant to document CD/CW/WP.213 of 19 September 1988.

GENERAL APPROACH:

1. OBJECTIVES

The purpose of this paper is to identify certain problems encountered during the NTI process and to evaluate different factors relating to such approach in particular:

- To evaluate whether the facility has produced any type of chemicals listed in schedule 2 not included in its declaration or whether it has diverted the produced chemicals to any prohibited purposes;

- To evaluate the cost of such an inspection;

- To determine physical constraints on inspection procedures;

- To evaluate the operational and economic impact of an inspection on a commercial facility;

- To evaluate the preparation needed for an inspection and to evaluate the minimum to fulfil the inspection mandate.

The experts of the Islamic Republic of Iran launched a national trial inspection in late June 1990 to assess the said elements at a facility producing dimethyl dichlorovinyl phosphate (DDVP) for production of household insecticides. The participation in this national trial inspection was also a good opportunity for various departments and organs in the Islamic Republic to get oriented with the Convention.

1.2 TYPE OF ON-SITE INSPECTION

The national trial inspection was carried out in accordance with document CD/CW/WP.213 and can be characterized as a "routine" inspection which included an initial visit.

1.3 ADVANCE INFORMATION

(a) Declaration

The full facility statistics and data were declared by the company to the authorized body in a specified format. The required data were in accordance with the declaration format set up in CD/961.

(b) Agreement on inspection procedure

A facility agreement was negotiated during the initial visit. Although the facility does not produce chemicals of schedule 2, but the model agreement contained in CD/961 served as the starting point for drafting the facility agreement with additions and changes required.

1.4 TYPE OF FACILITY TO BE INSPECTED

The facility inspected was a batch multi-purpose plant and a part of a chemical complex producing household and agricultural insecticides. The chemical produced was primarily organophosphorus used for formulating insecticides.

1.5 TYPE OF DECLARED ACTIVITY AT THE FACILITY

The declared activities at the facility were as follows:

- Consumption of trimethyl phosphate, schedule 3 chemical, and trichloroacetaldehyde for production of dichlorovinyl phosphate (dichlorovos);

- Production of dichlorovinyl phosphate (DDVP), as the key feed-stock for formulation of household and agricultural insecticides.

1.6 ACTUAL ACTIVITY AT THE FACILITY

During the inspection, all activities were going on normally. The declared facility consisted of raw material storage area (in liquid and solid form), production facility, waste treatment site, product storage area, formulating and packaging area and analytical area which were all operational and active.

2. DETAILED APPROACH:

2.1 THE INSPECTION MANDATE

A facility attachment was negotiated by the general manager of the Iranian National Industrial Organization (INIO) and the inspection. The facility attachment specified the areas to be inspected, the procedures of inspections, the route of inspection, sample taking points and procedures and the documents to be examined.

2.2 COMPOSITION OF THE INSPECTION TEAM

The inspection team consisted of 14 persons. Although the team was manned of more than needed but in a bid to show the importance of the Convention, representatives of concerned organs had also been invited.

The composition of the team was as follows:

1. An official from the Ministry of Foreign Affairs involved in CWC negotiations;

2. An official from the Joint Chief Staff Command of the Armed Forces Headquarters;

3. Three members of the Chemical Weapons Convention Committee in Teheran with PhD and MSC degrees in chemical engineering specialized in process engineering;

4. Representative of the Armed Forces in CW Committee in Teheran;

5. A representative from the Ministry of Industries in charge of verifying the facility's data documents;

6. Three experts from the Toxicology Research Department, University of Teheran, in charge of sample taking, transportation and analysis of samples;

7. Four experts from the Armed Forces (Revolutionary Guards Corps) specialized on chemical warfare detection.

2.3 INSPECTION EQUIPMENT

In a bid to further the efficiency of the inspection, some equipment was moved to the site including:

- An automatic detector designed by Iranian NBC Defence Establishment for detecting low concentration agents;

- Mobile "GC-Mass" spectrometer.

The instruments used for off-site analysis were:

- Gas chromatography (GC) and gas chromatography-mass spectroscopy (GC-Mass);

- Atomic absorption.

2.4 ACTIVITY PRIOR TO THE ARRIVAL OF THE INSPECTION TEAM ON-SITE

For preparation of the inspection, meetings were arranged with the authorities and visits were made to the central office of the company to obtain permission for the inspection, time of the initial visit and the inspection. The matters pertaining to facility arrangements were also discussed.

During the process of initial visit, facility declaration was made and facility attachment was signed. Preparatory work including visits mentioned earlier took a few weeks.

2.5 ADVANCE PREPARATION ON-SITE

Facility personnel served as informal guides and the management of the plant gave advance notification to make the inspection possible during the batch production in which a schedule 3 compound was produced. Arrangements were also made for transportation of off-site equipment. The office of the general manager served as the designated point of contact at the site.

2.6 DURATION OF INSPECTION AND INITIAL VISIT

- The initial visit took nine hours (27 May 1990);
- The facility agreement four man days (27 May 1990);
- The inspection 16 man days (27 June 1990);
- The preparation of the report 35 man days (15 July 1990).

2.7 MEASURES TO PROTECT CONFIDENTIAL INFORMATION

Based on the facility attachment, it was agreed that the information gathered from the facility or given by the management be treated as confidential (the result of the trial inspection in respect to the Convention is to be published in consultation with the management of the company and authorities concerned).

2.8 OPENING CONFERENCE

In the opening conference the leader of the inspection team introduced the members of this team, presented their credentials and outlined the inspection plan. The facility manager too introduced managers of departments and personnel available for inspection. He further outlined safety procedures for the inspectors. This conference took one hour in total.

2.9 PLANT ORIENTATION

During the initial visit, a plant orientation tour of the entire facility was arranged by the manager and the different sites of the facility were explained (the plant layout is found in the appendix).

2.10 INSPECTION OF AREAS AND FACILITY EQUIPMENT

The focal point of inspection was the DDVP reactor system and all equipment related to it including feed-stock storage and a variety of holding and storage tanks and pumps.

Actual size of reactors, vessels and tanks was verified with the help of physical measurements. Visual observations of raw material and product storage houses and tanks, analytical laboratory and waste treatment facility were made. In addition, samples were taken from the products in drums and from the waste treatment tanks and the reactor to verify the content.

The inspection team was split into five groups:

- Group I, responsible for sampling;
- Group II, responsible for verifying the process;
- Group III, responsible for process control analysis;
- Group IV, responsible for documentation analysis (records examination);
- Group V, observers.

During the course of the inspection, the leader of the team was responsible to meet the unforeseen needs of the inspectors with the aid of facility personnel.

2.11 INSPECTION OF OPERATION PROCEDURES

During the inspection, all the production and ancillary equipment was examined in detail to ensure their suitability for declared activity and their probable use for undeclared ones such as production of schedule 1 and other toxic chemicals. Notes were taken about the size of reactor in accordance with designed capacity and the physical characteristics of the reactor and the ancillary equipment. Particular attention was paid to the waste treatment and the safety measures in different areas. It was noted that the above mentioned factors were in accordance with the original design specification (the process diagram is presented in the appendix).

2.12 TYPES OF RECORDS NEEDED AND/OR AUDITED

Records and files of raw materials and products were checked to verify the consumption of raw materials used for production and the declared product. The examination process and the types of records studied are given in appendix 3.

2.13 SAMPLING AND SAMPLE TAKING PROCEDURES

The points of sample taking were specified in the facility attachment. Inspectors were equipped with the equipment and materials required for sampling. Sampling was carried out by the personnel in the presence of the inspection team. The areas and places from where the samples were taken were:

- Raw material storage tanks, vessels and sacks;
- Raw materials holding tanks to the reactor;
- Reactor (beginning, mid and end of reaction);
- DDVP carrying pumps. These pumps were washed by organic solvents and a sample was taken from the resulting solution;
- Waste treatment facility. Samples were taken from pipes carrying, washing liquid from reactor and tanks;
- Samples were taken from waste treatment tanks;

- Soil around raw material and product storage tank;

- Air samples from the reactor, waste treatment and the product storage areas.

2.14 SAMPLE HANDLING

All samples which were taken in two sets, were sealed, numbered and coded. One set was left in the facility and another was taken for off-site analysis. Someone from the facility was assigned to accompany the samples to the off-site laboratory to observe the analysis process. Due to the far distance of the facility from the laboratory, and because of high temperature, a portable cold storage box was used to prevent any outside effect.

2.15 ANALYSIS OF SAMPLES

Samples taken from feed-stocks, reactor solution and products were analysed on-site and in presence of the inspectors. Another part of these samples was analysed off-site (in the presence of facility personnel). Samples from soil, pumps, waste and air were also analysed off-site.

The results of such analysis proved that no other chemicals were used or produced in addition to the declared activities.

2.16 TYPES OF ANALYSIS

The analysis carried out to verify the activities in accordance to the declared format proved that no chemicals of schedule 1 had been produced. For this purpose, a gas chromatography (GC) and a gas chromatography-mass spectroscopy (GC-MASS) were used. Non-presence of schedule 1 chemicals was verified by military detection unit.

2.17 DOCUMENTATION OF THE INSPECTION

The plant layout, the equipment layout, the piping plan and the electrical plan were presented during the initial visit.

The information collected during the initial visit and the reports prepared by different inspection groups were filed in the chemical weapons defence establishment.

2.18 EVALUATION BY INSPECTORS

The inspectors carefully evaluated the declared data with the actual activities in the facility. The difficulties and obstacles during the inspection process were analysed by the inspectors and suggestions were made.

2.19 CLOSING CONFERENCE

During the one-hour closing conference, the data and the information collected were discussed and confidentiality of the facility attachment was underlined.

2.20 ANOMALIES, DISPUTES AND COMPLICATIONS

- The formulation of the final product and activities was not disclosed.

- Safety instructions and procedures were practised properly.

- The efficiency of the reactor was discussed.

2.21 REPORT OF THE INSPECTION TEAM

During the inspection, the inspectors collected the data in note forms. However, since some samples had to be taken for off-site analysis, a time lag of four days was needed for submission of the report. Appendix 2 is the extract of the inspectors' report.

2.22 THE IMPACT OF THE INSPECTION ON THE FACILITY OPERATIONS

In the facility attachment, the inspection route and timing was planned in such a way that the inspection took place from the beginning of the batch in order to take the shortest possible time for inspection. During the inspection, the managing director accompanied the leader of the inspection team and the department managers assisted the inspectors.

OTHER MATTERS (A)

COSTS

The costs of routine inspection varies considerably from one country to another. This is mainly due to the labour, accommodation, laboratory and transportation costs. The break down of the inspection procedures carried out and their costs, are as follows:

- Initial visit preparation and initial visit, 1.5 man months;

- Pre-inspection preparation and the inspection 1.5 man months;

- Laboratory, 800,000 Rials;

- Report writing, 1.5 man months;

- Transportation: the cost of transportation depends on the location of the facility.

  The costs of transportation for this exercise were around 250,000 Rials (70 Rials = $1);

- Other expenses such as meals etc., were around 150,000 Rials.

Hence the total cost of the routine inspection was around 3 million Rials which is equivalent to about $42,000.

For the present NTI exercise, no accommodation was needed, but under the assumption of an average 6 man is needed for the inspection to be carried out in four days, then the cost of accommodation would be around 500,000 Rials or about $7,200.

It is to be noted that the cost of military equipment and instruments were not considered.

RESULTS

The results obtained by the national trial inspection (NTI) exercise carried out in one of the chemical plants producing DDVP (a non-schedule chemical) by using trimethyl phosphate (a schedule 3 chemical) show that:

- The process was in accordance to what was declared;
- No other chemicals except those declared, were found in analysis of the samples;
- No warfare agent was detected by military equipment, on-site mobile GC/MS or the analysis carried out in off-site;
- No considerable discrepancies were experienced during the process of records analysis.

Organizations co-operated in this exercise were highly qualified and experienced, due to valuable experiences they gained during the war. The presence of very accurate analytical instruments also prevented any substantial error.

It is worth mentioning that for the double check of the analytical results, a random set of samples was sent to the Chemistry Department of the University of Tabriz in the north-west of the country.

With regard to the cost of such an inspection, it should be mentioned here that such an inspection costs between $50,000 to $60,000.

DISCUSSIONS AND RECOMMENDATIONS

The present national trial inspection carried out by the Islamic Republic of Iran is in accordance to the proposals made by the Ad Hoc Committee on Chemical Weapons, in order to verify whether it is possible to ascertain that the declared chemical industry facilities are not used for production of chemicals prohibited by the CW Convention. The aim of the inspection measures is to create confidence among the parties to the convention and to envisage, at this stage of negotiation, the conditions in which the highly complex tasks of the team of inspectors could be carried out. Although many countries have already indicated, by their national trial inspection exercises, the national problems of such an inspection, but since the task is not easy, it requires a more clear definition of the extent of the work to be done, the responsibility of each member of the team and the role of the facility representative in prepared formats.

The importance of such inspections are known to every party, and since the results obtained by the routine inspection and even challenged inspection can be very vital for them, hence the inspection must be carried out in a carefully designed manner.

Based on the experience gained by the Iranian experts, the following conclusions and recommendations are presented:

1. Iran is a big country and access to plants needs a relatively long time. This fact makes timing and adequate preparations necessary.

2. In the developing countries, means of communications for optimal inspection should be taken into account.

3. The routine inspection is applicable.

4. Trained and experienced personnel can shorten the inspection time considerably and hence the inspection procedures.

5. Prior to the entry into force of the CWC, the chemical industries must be acquainted with the convention through technical seminars and meetings.

6. The high cost of the routine inspection show that to maintain the cost in optimized level a priority categorization method should be developed to inspect the chemical industries more effectively and less time consuming.

7. For reduction of the inspection time, military equipment and instruments especially detection units and kits can play an important role and the cost of the inspection can considerably be reduced. Application of military instruments in inspections are therefore recommended.

APPENDIX 1

PROCESS INSPECTION

The inspection and verification of the process equipment were thoroughly carried out by two process technologists for three hours.

For the production of Dichlorovos, trichloroacetaldehyde and three methyl phosphite are reacted according to the following chemical formula:

$$(CH_3O)_3P + CCl_3CHO \xrightarrow{CH_3Cl} H_3C\text{-}O\text{-}P\text{-}O\text{-}CH = CCl_2 + H_3C\text{-}O \quad \text{(with O on P)}$$

The raw materials and the solvent from the holding tanks are transferred to the reactor. The low boiling point gases are discharged from the top of the reactor, to the heat exchanger and finally to the solvents separator vessels.

The impure product is purified by a distillation column. The purified Dichlorovos is finally transferred to the storage vessels. In every batch, 221 Kilos of the product is produced.

The process inspectors noted the following:

- The signs, physical conditions, mode of operations and the flows of each processes equipment.
- The capacity and the physical conditions of the raw materials storage vessels and the quantity of raw materials in storage.
- The thorough put and the physical conditions of the pipes and pumps.
- The specifications of the equipment and the means of heating and cooling of the materials.
- The specifications of the waste disposal equipment.
- The signs and capacities of product storage vessels.

The result of the process inspection was in accordance with the declared activity and no considerable discrepancies were detected.

The flow diagram of the process is given in Figure (1). Also in this diagram the points of sample taking are shown.

APPENDIX 2

SAMPLE TAKING AND ANALYSIS

For the analysis of the samples, three analytical chemists with Ph. D degrees specialized in toxicology were appointed. The samples were taken by the facility personnel under the supervision of the chemists.

It is worth mentioning that the points of sample taking were specified before the inspection proceeded.

The samples were taken in double by appropriate means and equipment such as sampling pumps.

The points where examples were taken were as follows:

- Raw material storage vessels and holding tanks.

- Reactor, before the initiation of the reaction, half time of the completion of the reaction and in the end of the batch time by using the sampling valve attached to the reactor.

- The dust on the filters of masks (in random selection) and the air conditioning system.

- Condensate of the reaction gases from the heat exchanger.

- The bottom flow and the upper flow of the distillation column.

- Pumps connected to the reactor before and after the operation, by washing with solvent. Because of the batch operation of the process, sample taking from the pumps did not disturb the production programme of the facility.

- Dust around the reactor area.

- Waste treatment area; (washing solvent and sludge). The sample taking was done by floating the sample jar.

  Soil around the waste treatment area.

- Dust on the filter of masks (in random manner selection) and air conditioning system.

- Product and byproduct storage vessels and drums (random order).

ANALYSIS

Instrumental analysis was carried out for all samples including DDVP, to detect the purity compounds.

Instruments which were used are:

- Gas chromatograph (GC).

- Mass-spectrometer connected with gas chromatograph (GC-MS).

- Nuclear magnetic resonance (NMR).

The samples were transported by using air tight glass bottles and portable cold boxes.

All the samples were labeled and a copy of the results of analysis was sent to the facility.

APPENDIX 3

DOCUMENTATION INSPECTION

For the examination of records two accountants were appointed. To achieve the best results, it is suggested that the qualification of industrial accountant is necessary. The records which were examined during the inspection period for three hours are as follows:

- Feed-stocks including quantity in storage and the quantity used in the process and the quantity received.

- End product and byproduct records including quantity of output and quantity delivered to the users and the quantity in storage.

- Records and analytical results from waste treatment analysis.

- Records of the purity analysis of the raw materials and the product.

- Records of the quantity of waste resulting from the process.

- Examination of the raw material requires form, price and sales records.

- Batch times.

- Examination of the measuring instruments records.

- Examinations of the utilities consumption (no reliable records were kept).

For the analysis of the above factors, the records of a period of three months were available and no records were removed from the facility.

LANGUAGE

It is to be noted that most of these records were in Persian.

APPENDIX 4

UTILITIES

For the inspection of the utilities production department, two chemical Engineers were appointed.

- The location of the units and the equipment were checked against the documents provided in the initial visit.

- The utilities consumption figures from the instruments were recorded.

The water consumption figure was not reliable due to the failure of the instruments. The sign and capacities of the equipment used for water purification systems.

APPENDIX 5

MILITARY EQUIPMENT

For the detection and monitoring of the existence of toxic warfare agents, a team of four from NBC defence establishment together with their appropriate instruments were appointed. They monitored the air in the storage area, reactor area, separation area, product holding tanks area and the product storage area. For such purpose they employed four individual and different pieces of equipment.

The results of these examinations were provided in the appropriate forms.

The information included in the forms are: the site of sampling, the time, the chemical to be detected, the name of the equipment tested, the time duration for revealing results, the humidity, the quantity of the chemical in the air, in ppm, the temperature of the site and the pressure of the air.

In these experiments, the non-existence of Nerve Gas warfare agents was to be verified.

APPENDIX 6

SAFETY MEASURES

During the inspection, the inspectors observed the safety rules and regulations adopted at the facility and checked them against the standard rules. Also the workers in the process area, raw material storage area, product storage area were interviewed about the possible sicknesses in the past and also a sample of their blood were taken for further examination off-site. These results were available in four days. The colinstrasse of the worker's blood was checked by using appropriate kit. The individual safety measures applied in the facility against gases and particles were analyzed. There was no continuous monitoring system for toxic gases in the facility area.

Also some filters from air conditioning system were taken to be analyzed off-site. The medical records of the workers were examined and no misleading absence was observed.

RESULTS

The results obtained by the National Trial Inspection (NTI) exercise carried out in one of the chemical plants producting DDVP (a non schedule chemical) by using trimethyl phosphite (a schedule [3] chemical) show that:

- The process is in accordance to what it was declared.
- No other chemicals except those in declaration, were encountered during the analysis of samples.
- No warfare agent was detected on site mobile GC/MS and the analysis carried out off-site by the military equipment.
- No considerable discrepancies were encountered during the analysis of records.

FIG(1). PROCESS DIAGRAM OF DDVP PRODUCTION UNIT